APR 27 1992

Don Kampbell, Research Chemist
U. S. Environmental Protection Agency
Robert S. Kerr Environmental Research Laboratory
PO Box 1198
Ada, OK 74820

Re: L. E. Carpenter Company (aka Dayco Corporation) Site in Wharton, NJ

Dear Dr. Kampbell:

I have had a chance to review your March 1992 report entitled "Biotreatability of a Site Soil Contaminated with Xylene and Dioctyl Phthalate." I was pleased to read that even the most contaminated soil sample from the L. E. Carpenter Company site showed significant biological activity.

As discussed with you on April 23, 1992, I have some comments on the report which I am enclosing in the form of notes handwritten on a copy of the report. My comments deal mainly with providing some additional details so that the research procedures and findings will be more clearly understood. After consulting with the New Jersey Department of Environmental Protection and Energy (NJDEPE), I am not expecting any additional comments from that agency.

Another EPA Region II reviewer prepared a list of eight comments on the report. I have enclosed those comments with some minor editorial changes. Please consider whether these comments merit any changes in the report.

In addition, I am interested in how the cleanup goals for bis(2-ethylhexyl)phthalate (DEHP) may affect the total cleansing times. The State of New Jersey has recently proposed Cleanup Standards for DEHP, among other pollutants. The proposed DEHP Cleanup Standards are 49 mg/kg, 100/mg/kg and 210 mg/kg for residential surface soils, residential subsurface soils and non-residential soils, respectively. I believe that the subsurface standard would control the cleansing time since the most contaminated soil at the site tends to be near the water table, about five feet below the surface. There is currently some doubt as to whether the residential or non-residential standards will be the goals for this site. Since this is a practical matter rather than a research question, it needn't be addressed in the text of your research paper but may be addressed separately.

JJ 4/27

I don't intend to distribute the report to the site owner or to anyone outside of EPA and NJDEPE until you have advised me that the report is in final form and may be released.

Please contact me at (212) 264-8098 if you wish to discuss this matter.

Sincerely yours,

Jonathan Josephs, Project Manager
New Jersey Superfund Branch II
Emergency and Remedial Response Division

Enclosure

cc: C. Purcell, NJDEPE

bcc: D. McChesney, DGWP
M. Breville, ORD

Comments on the March 1992 Report on the Biotreatability of a Site Soil Contaminated with Xylene and Dioctyl Phthalate

1. Why weren't the site soil samples used to estimate biodegradation potential? Since the site soils contained culturable bacteria and the array of contaminants present at the site, this may have been the most definitive methodology.

2. Was oxygen added as a supplement to the microcosms? If not, could oxygen have become rate limiting? Could the JP-4 and xylene cultures have become anaerobic? Was the water oxygenated or deoxygenated?

3. NPK is shown as a supplement for the JP-4 and Xylene treatments shown in Table 4. It is assumed that this is nitrogen, potassium, and phosphate, but there is no mention of nutrient additions in the text. While the analytical results suggest sufficient nutrient concentrations in the soil samples, the bio-availability of the nutrients could be an issue. Was the water supplement deionized? If not, what were the concentrations of NPK?

4. The rubicon sand with which the DEHP spiked microcosms were run was probably inoculated with microorganisms. What was the source of the microbial seed? Were they from a pure culture? Were they acclimated to DEHP? Were they aerobic bacteria or some other microorganisms? Did the organisms exhibit population enhancement between the beginning and the end of the experiment? These issues could highlight the most significant limitations of the study.

5. Was there a sterile control?

6. Is the data shown in Figure 4 a mean value for all of the microcosms, and if so, what was the range and standard deviation? Since relatively small differences were seen between the water, JP-4, and xylene treatments, this data could help define whether the differences are real or the result of experimental variation. Also, were any replicate samples run? How did they agree?

7. Does examination of the GC results show the presence of any peaks which may be reaction products? Did the relative heights of such peaks change over the course of the experiment? Did any other compounds present in the samples show evidence of biodegradation?

8. Why were xylene and JP-4 chosen as culture supplements? Since biodegradation by methanogens of a wide variety of compounds has been demonstrated, why wasn't methane tried?

BIOTREATABILITY OF A SITE SOIL CONTAMINATED WITH XYLENE AND DIOCTYL PHTHALATE

Don H. Kampbell, USEPA/RSKERL

Dennis D. Fine and Jerry W. Anderson
ManTech Environmental Technology

See the next page for comments on the chemical nomenclature.

March 1992

There are inconsistencies in the terms used to refer to bis (2-ethylhexyl) phthalate and related compounds. For example:

- page 1 refers erroneously to "octyl phthalate."
- the abbreviation "DEHP" should be used to refer only to bis (2-ethylhexyl) phthalate (aka, di (2-ethylhexyl phthalate)
- on page 2, "DEHP" apparently refers to bis (2-ethylhexyl) phthalate, yet at the top of page 3 it appears to include related compounds from the bottom of Table 2 (e.g., diisooctyl phthalate)
- In Table 2, "Dioctylphthalate" and "Dioctyl phthalate" are listed as two different compounds. This is confusing.

The nomenclature should be revised in consultation with an organic chemist.

Introduction

A large number of organic compounds can be mineralized or transformed through microbiological processes. Degradation rates vary for different compounds and are influenced by variables such as concentration, nutrients, moisture, aeration, temperature, degree of acclimation, plus many others. In situ biodegradation as a cleansing technology has the attribute of being a ~~natural~~ process of nature.

A site was used for over 40 years to manufacture vinyl wallcovering. Chemical spillage and waste disposal has contaminated portions of the site with metals and various organic chemicals, predominately xylene and octyl phthalate.

Treatability studies were conducted using laboratory techniques on vertical profile core samples obtained from the ~~manufacturing~~ site. The objective of the study was to ~~show that~~ determine whether in-situ biodegradation ~~was~~ is a feasible alternative for remediation of the site.

Experimental Methods

Vertical profile core samples were collected to a depth of eight feet by Ken Tyson of Weston Consultants at the L.E. Carpenter site near Wharton, New Jersey on October 24-25, 1991. These locations were sampled: TS-01 = no contamination; TS-02 = moderate contamination; TS-03 = high contamination. Reference map. The core samples representing three two-foot increments were placed in glass pint jars and shipped to our facility. The nine samples were analyzed by standard tests as shown in Table 1. A mixture of the three depth core samples from TS-03 location was extracted with methylene chloride for organic compounds identification by a gas chromatography/mass spectrometer method. Oxygen and carbon dioxide were

measured by removing aliquots of headspace gas from 160 ml. microcosm glass serum bottles containing ten percent by volume core material and capped with teflon-coated butyl rubber septa. Core material used for testing was separated from coarse sand and pebbles which were about ⅓ the total mass of core material.

Determination of rate of dioctyl phthalate (DEHP) was done by adding 50 grams of air-dry Rubicon Sand soil to replicate 160 ml. serum bottles. The air-dry soil contained 2.8 percent moisture. Four treatments used were as follows: 1 = air-dry soil as a control; 2 = nine percent by weight water was added; 3 = above water + 1000 ul JP4 jet fuel fumes; 4 = above water + 600 ul xylene mixture vapor. The air-dry soil in each bottle contained 0.5 grams DEHP. The microcosms were acclimated for two weeks then initial treatment sets were extracted with methylene chloride for analysis of DEHP by a gas chromatography method. A second treatment set was extracted and analyzed 42 days later.

Table 2 refers to GC/MS. Is this the same method?

Results and Discussion

Nutrient requirements most limiting to microbiological processes in soils are nitrogen and phosphorus. All nine of the core samples tested contained sufficient nitrogen and phosphorus (Table 1). Bacterial cell counts for a viable soil are usually in the 10^7 to 10^8 range. The site core samples had a total cell count indicative of vigorous bacterial activity. Dehydrogenase activity also indicated the presence of high viability and the absence of toxicological restraints.

Chemical analyses confirmed that the magnitude of dioctyl phthalate contamination at the three different coring locations was low, moderate, and above moderate. Moisture contents of the core materials ranged from 10 to 32 percent.

Moisture content values should be tabulated

Dioctyl Phthalate represented 95 percent of the contaminants in a mixture of cores from the above moderate location (Table 2). Other components such as the volatile aromatic hydrocarbons may vary at different locations and depths depending on losses by emissions, degradation, dissolution by soil water, and original concentrations.

Active microbiological processes typically involve oxygen and carbon dioxide especially under aerobic conditions. Data recorded in Table 3 was generated by core material microcosms for different 22°C incubation time periods. A definite response occurred for oxygen consumption and carbon dioxide generation for the contaminated core materials. The trend of the data indicated that the rate of carbon dioxide generation was very close to the rate of oxygen consumption.

An estimation of degradation rate of DEHP in soil was determined with sandy soil microcosms as listed in Table 4. Biodegradation by the control was limited by lack of soil moisture. Water and xylene vapors when present accelerated the rate of DEHP biodegradation. The data indicated that about 0.1 gram DEHP per kilogram soil per day was biodegraded. If a mass balance process could logically be extrapolated from lab studies to actual field conditions, the total cleansing time period for in-situ biodegradation would be 1440 days (3 years) and 4380 days (12 years) for moderate and greater than moderate locations at the field site, respectively.

Conclusion

The soil microcosm laboratory studies showed that cored material contaminated with the L.E. Carpenter site waste was being naturally remediated. The rate of biodegradation was estimated to be 0.1 milligram DEHP per day per

How extrapolated? What was the cleanup goal?

Was this the maximum estimated rate? How determined?

kilogram soil. Actual field rates can vary by orders of magnitude dependent on controlling factors. The lab studies did determine that the waste contaminated core materials are biologically active.

A potential for in-situ bioremediation exists at the site. Possibilities exist to enhance the waste biotransformation process by installation of a bioventing treatment system. Development of a productive unit process would require further effort in lab studies, a pilot plant, literature search, and experienced personnel.

Table 1 - Dioctyl Phthalate, Nitrogen, Phosphorus, DHA, and Bacteria Cell Count for Core Samples from L.E. Carpenter Site

Sample	Depth Feet	AODC cells/gm $X10^6$	DHA formazan µg/gm	Dioctyl phthalate mg/Kg	Nitrate & Nitrite mg/Kg	Total Phosphorus mg/Kg	Total Kjeldahl Nitrogen mg/Kg
TS-01-01	2-4	34	3.7	8	6.5	432	497
TS-01-02	4-6	15	2.2	7	7.8	428	298
TS-01-03	6-8	14	3.6	4	11.	379	807
TS-02-01	2-4	25	21.	144	8.5	1060	665
TS-02-02	4-6	30	58.	139	1.2	941	1510
TS-02-03	6-8	53	31.	20	32.	731	848
TS-03-01	2-4	8	22.	152	3.9	722	1510
TS-03-02	4-6	14	6.6	438	1.2	573	2030
TS-03-03	6-8	7	2.5	410	1.2	638	1980

reference ~~test~~ methods

Table 2 - Relative Abundance of Major Components In a Mixture of Cored Material from TS-03 Location

Component	Relative Peak Area by GC/MS
Ethylbenzene	0.18
m+p-Xylene	1.10
o-Xylene	0.21
Decane	4.96
Trimethyldecane	4.01
Ethylmethylheptane	1.52
4,7-Dimethylundecane	0.74
Octafluoronaphthalene (I.S.)	1.00
Unknown Phthalate	0.09
Butyl 2-methylpropylphthalate	0.52
2-Ethylhexyldiphenyl phosphate	3.75
Dioctylpthalate	0.79
Diisooctylphthalate	91.98
Bis(2-ethylhexyl)phthalate or Dioctyl phthalate	241.45

Is this di-n-octyl phthalate ?

Reference test method

Concentrations should be tabulated to enable comparison to other site data

Table 3 - Microcosm Headspace Oxygen and Carbon Dioxide

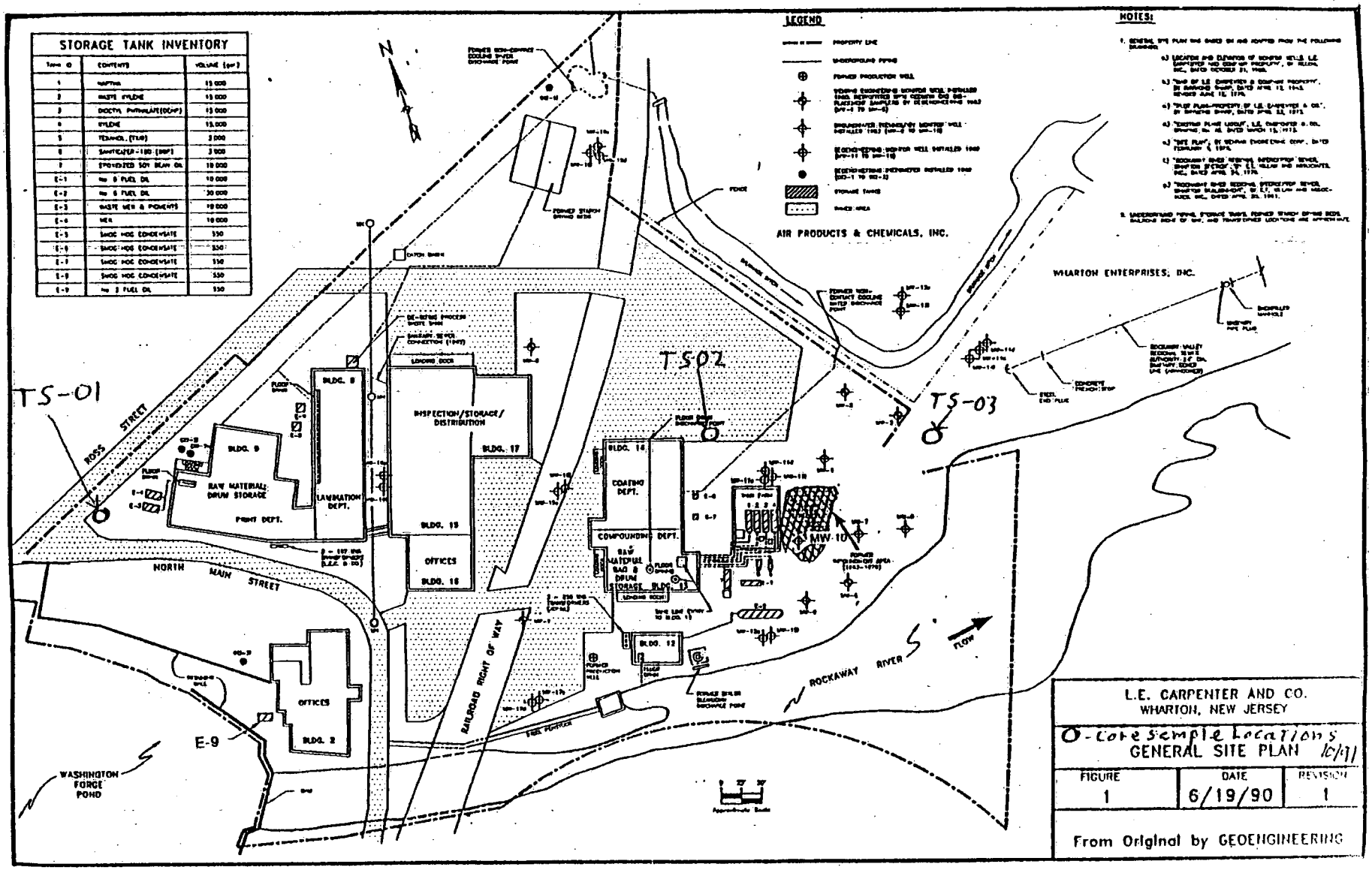
Sample	Time Period, Days							
	2	5	17	24	2	5	17	24
	Oxygen, %				Carbon Dioxide, %			
TS-01-01	20.8	19.9	21.2	18.9	0.68	0.62	1.10	1.8
TS-01-02	20.7	19.9	21.1	18.9	0.70	0.71	1.10	1.6
TS-01-03	21.0	20.0	21.2	19.2	0.65	0.61	0.94	1.6
TS-02-01	18.1	16.7	10.5	7.5	3.40	3.20	8.00	10.1
TS-02-02	12.2	9.1	1.4	1.1	6.30	7.60	14.00	15.2
TS-02-03	16.2	14.1	9.1	6.0	4.70	5.40	9.40	11.6
TS-03-01	18.3	17.2	14.3	11.2	3.30	3.10	5.80	7.6
TS-03-02	6.7	8.6	1.0	1.1	9.00	8.40	14.70	15.6
TS-03-03	7.8	5.4	2.4	1.4	9.20	11.00	14.10	16.0

Reference test methods

Table 4 - Degradation of DEHP by Rubicon Sand Microcosms During 42 Days Incubation at 22°C

Microcosm	**Amount DEHP Utilized from 10 gm Added/kg Soil**
Air-dry Control	1.0
Water Added	4.3
Water + NPK + JP4 Added	4.4
Water + NPK + Xylene Added	5.5

Was "NPK" explained anywhere in the report? What is it?



Tank ID	CONTENTS	VOLUME (gal)
1	NAPTHA	15 000
2	WASTE XYLENE	15 000
3	DIOCTYL PHTHALATE (DOP)	15 000
4	XYLENE	15,000
5	TOLUENOL (TOL)	2 000
6	SANITIZER-100 (BBP)	2 500
7	EPOXIDIZED SOY BEAN OIL	10 000
E-1	No. 6 FUEL OIL	10 000
E-2	No. 6 FUEL OIL	30 000
E-3	WASTE MEK & PGMEHYD	10 000
E-4	MEK	10 000
E-5	SWDC HOC CONDENSATE	550
E-6	SWDC HOC CONDENSATE	550
E-7	SWDC HOC CONDENSATE	550
E-8	SWDC HOC CONDENSATE	550
E-9	No. 2 FUEL OIL	550